Aspirin Boluses

DEFINITION
Aspirin Boluses contain NLT 90.0% and NMT 110.0% of the labeled amount of aspirin ($C_9H_8O_4$).

IDENTIFICATION
- **A. PROCEDURE**
  Analysis: Crush 1 Bolus. Boil a portion of the powder, equivalent to 300 mg of aspirin, with 50 mL of water. Cool and add a drop of ferric chloride TS.
  Acceptance criteria: A violet-red color is produced.
- **B.** The retention time of the aspirin peak of the *Sample solution* corresponds to that of the *Standard solution*, as obtained in the *Assay*.

ASSAY

Change to read:

- **PROCEDURE**
  Mobile phase: 2 g/L of sodium 1-heptanesulfonate in a mixture of acetonitrile and water (15:85). Adjust with glacial acetic acid to a pH of 3.4.
  Diluent: Acetonitrile and formic acid (99:1)
  Standard solution: 0.4 mg/mL of USP Aspirin RS and 0.01 mg/mL of USP Salicylic Acid RS in *Diluent*
  Sample stock solution: Nominally 4 mg/mL of aspirin prepared as follows. Finely powder NLT 10 Boluses. Transfer a portion of the powder to an appropriate volumetric flask and dilute with *Diluent* to volume. Stir the solution by mechanical means for 15 min.
  Sample solution: Nominally 0.4 mg/mL of aspirin from *Sample stock solution* in *Diluent*. Pass a portion of this solution through a filter of 0.5-µm or finer pore size. Use the filtrate. (IRA 1-Jan-2017)
  Chromatographic system
  (See *Chromatography* ⟨621⟩, *System Suitability*.)
  Mode: LC
  Detector: UV 254 nm
  Column: 4.6-mm × 25-cm; 5-µm packing L1
  Flow rate: 1 mL/min
  Injection volume: 20 µL
  System suitability
  Sample: *Standard solution*
  [NOTE—The relative retention times for salicylic acid and aspirin are 0.6 and 1.0, respectively.]
  Suitability requirements
  Resolution: NLT 2.0 between salicylic acid and aspirin (IRA 1-Jan-2017)
  Relative standard deviation: NMT 2.0% for aspirin
  Analysis
  Samples: *Standard solution* and *Sample solution*
  Calculate the percentage of the labeled amount of aspirin ($C_9H_8O_4$) in the portion of Boluses taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response of aspirin from the *Sample solution*
r_S = peak response of aspirin from the *Standard solution*
C_S = concentration of USP Aspirin RS in the *Standard solution* (mg/mL)
C_U = nominal concentration of aspirin in the *Sample solution* (mg/mL) (IRA 1-Jan-2017)

Acceptance criteria: 90.0%–110.0%

PERFORMANCE TESTS

Change to read:

- **DISSOLUTION** ⟨711⟩
  Medium: 0.5 M phosphate buffer (see *Reagents, Indicators, and Solutions—Buffer Solutions*), pH 7.4; 900 mL
  Apparatus 2: 75 rpm
  Time: 45 min
  Diluent: Acetonitrile and formic acid (99:1)
  Standard solution: USP Aspirin RS in *Diluent* (IRA 1-Jan-2017) at a suitable concentration. [NOTE—Prepare the solution at the time of use.]
  Sample solution: Pass a portion of the solution under test through a suitable filter. Dilute with *Diluent*, if necessary.
  Instrumental conditions
  Mode: UV-Vis
  Analytical wavelength: The isosbestic point of aspirin and salicylic acid at 265 ± 2 nm
  Analysis
  Samples: *Standard solution* and *Sample solution*
  Calculate the percentage of the labeled amount of aspirin ($C_9H_8O_4$) dissolved:

$$\text{Result} = (A_U/A_S) \times C_S \times V \times D \times (1/L) \times 100$$

A_U = absorbance of the *Sample solution*
A_S = absorbance of the *Standard solution*
C_S = concentration of the *Standard solution* (mg/mL)
V = volume of *Medium*, 900 mL
D = dilution factor of the *Sample solution*, if necessary
L = label claim (mg/Bolus) (IRA 1-Jan-2017)

  Tolerances: NLT 80% (*Q*) of the labeled amount of aspirin ($C_9H_8O_4$) is dissolved.
- **UNIFORMITY OF DOSAGE UNITS** ⟨905⟩**:** Meet the requirements

IMPURITIES

Change to read:

- **LIMIT OF SALICYLIC ACID**
  Mobile phase, Diluent, (IRA 1-Jan-2017) **Sample solution, Chromatographic system,** and **System suitability:** Proceed as directed in the *Assay*.
  Salicylic acid standard solution: 0.01 mg/mL of USP Salicylic Acid RS in *Diluent* (IRA 1-Jan-2017)
  Analysis
  Samples: *Sample solution* and *Salicylic acid standard solution* (IRA 1-Jan-2017)
  Calculate the actual concentration (*C*), in mg/mL, of aspirin ($C_9H_8O_4$) in the *Sample solution* taken:

$$\text{Result} = C_U \times (F/100)$$

C_U = nominal concentration of aspirin in the *Sample solution* (mg/mL)
F = percentage of the labeled amount of aspirin ($C_9H_8O_4$) in the portion of Boluses taken, as determined in the *Assay*

  Calculate the percentage of salicylic acid ($C_7H_6O_3$) in the portion of Boluses taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C) \times 100$$

r_U = peak response of salicylic acid from the *Sample solution*
r_S = peak response of salicylic acid from the ●*Salicylic acid standard solution*● (IRA 1-Jan-2017)
C_S = concentration of USP Salicylic Acid RS in the ●*Salicylic acid standard solution*● (IRA 1-Jan-2017) (mg/mL)
C = actual concentration of aspirin in the *Sample solution*

Acceptance criteria: NMT 0.3%

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight containers.
- **LABELING:** Label Boluses to indicate that they are for veterinary use only.

- **USP REFERENCE STANDARDS** ⟨**11**⟩
  USP Aspirin RS
  USP Salicylic Acid RS